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(71) Applicant (*for all designated States except US*): **THE LUBRIZOL CORPORATION** [US/US]; 29400 Lakeland Blvd., Wickliffe, Ohio 44092-2298 (US).

(72) Inventors: **BURRINGTON, James D.**; 6858 Bramblewood Lane, Mayfield Village, Ohio 44143 (US). **GEORGE, Herman F.**; 11840 Clarkwood Lane, Chardon, Ohio 44024 (US). **MARTIN, John R.**; 6931 Bunbury Lane, Concord Township, Ohio 44077 (US). **KORNBREKKE, Ralph E.**; 8340 Tulip Lane, Chagrin Falls, Ohio 44023 (US).

(74) Agent: **GILBERT, TERESAN W. à**; The Lubrizol Corporation, Patent Dept./Mail Drop 022B, 29400 Lakeland Blvd., Wickliffe, Ohio 44092-2298 (US).

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(54) Title: GELS THAT REDUCE SOOT AND/OR EMISSIONS FROM ENGINES

(57) Abstract: A soot reducing media that reduces the soot content in lubricating oil in an engine. Further a process employing a gel to decrease the amount of soot in the lubricating oil of an engine and/or decrease the emissions from an engine.

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Title: GELS THAT REDUCE SOOT AND/OR EMISSIONS FROM ENGINES

Background of the Invention

The present invention relates to a novel gel composition that results in the decrease in the amount of soot in a lubricating oil in an engine and/or decrease the amount of emissions particularly soot, hydrocarbons and/or nitrogen oxides (NO, NO_2, N_2O, collectively known as NOx) from an engine.

Soot may be present in any lubricating oil used in a lubrication system of any engine that generates soot such as internal combustion engines, spark ignited engines, stationary engines, off and on highway engines and the like. Internal combustion engines, in particular diesel fueled engines, generate carbonaceous soot particles. During combustion the fuel is injected into the combustion chamber in the form of small droplets. During the combustion process, soot particles form from incompletely combusted fuel. The lubricating oil for the cylinders and the rings contain the soot from the incomplete combustion. As the pistons move up and down in the chamber, the soot particles that have formed go into the lubricating oil system of the pistons, rings, through the cylinder and into the reservoir. Accordingly, the generated soot in the engine oil contributes to problems with engine lubrication.

Soot is also a problem in modern diesel engines with fuel injection systems. The fuel injection system has been designed to produce less emissions, but has increased the formation of soot in the lubricating oil of the engine. It further requires more frequent oil change intervals to prevent the concentration of soot particles in the oil from exceeding acceptable limits.

The suspended soot particles in the lubricating oil have the effect of increasing the viscosity and creating wear particles in the lubricating oil. Accordingly, the soot acts like an abrasive and induces wear in the engine parts. Further, high soot levels result in shorter drain intervals and more oil changes.

Dispersants have been used in lubricating oils to suspend the soot build up so as to reduce the detrimental effects of the soot on engine wear. However, an oils' capacity to protect an engine is limited, even with the dispersants. In addition, soot

emissions in particular soot, hydrocarbons and/or Nox from an engine. The engines that can use the gel include, but are not limited to internal combustion engines, stationary engines, generators, diesel and/or gasoline engines, on highway and/or off highway engines, two-cycle engines, aviation engines, piston engines, marine engines, railroad engines, biodegradable fuel engines and the like. In one embodiment the engine is equipped with after treatment devices, such as exhaust gas recirculation systems, catalytic converters, diesel particulate filters, NOx traps and the like.

Detailed Description of the Invention

In accordance with the present invention the soot concentration is decreased from a lubricating oil in an engine thereby avoiding the deleterious effects on the engine from the soot, including viscosity, wear and emissions. Furthermore, the emissions of an engine is decreased thereby improving the environment.

The soot level is reduced by contact with the gel. The gel is positioned within the lubricating system, anywhere the gel will be in contact with the lubricating oil. The gel is positioned anywhere that the circulating oil contacts the gel such as full flow of oil, bypass of the oil in the reservoir or combinations therein. The location of the gel in the lubricating system includes but is not limited to a filter, drain pan, oil bypass loop, canister, housing, reservoir, pockets of a filter, canister in a filter, mesh in a filter, canister in a bypass system, mesh in a bypass system and the like. One or more locations can contain the gel. Further, if more than one gel is used it can be identical, similar and/or a different soot-reducing gel.

In one embodiment it is desirable to provide a container to hold the gel, such as a housing, a canister, a structural mesh or the like anywhere within the lubricating oil system, for example, a filter in a housing of an engine oil lubricating system. The necessary design feature for the container is that at least a portion of the gel is in contact with the oil.

In one embodiment, the gel is positioned anywhere in the filter. The filter is a desirable location to place the gel because the gel and/or spent gel can easily be removed, and then replaced with a new and/or recycled gel.

Component A includes but is not limited to antioxidants; dispersants; ashless dispersants such as Mannich dispersants; succinics; esterfied maleic anhydride styrene copolymers; maleated ethylene diene monomer copolymers; surfactants; emulsifiers; functionalized derivatives of each component listed herein and the like; and combinations thereof. Component A can be used alone or in combination. In one embodiment the preferred A is polyisobutenyl succinimide dispersant.

Component B includes but is not limited to dispersants, detergents, overbased detergents, carbon black, silica, alumina, titania, magnesium oxide, calcium carbonate, lime, clay, zeolites and the like; and combinations thereof. Component B can be used alone or in combination. In one embodiment Component B is an overbased alkybenzenesulfonate detergent.

Component C includes but is not limited to the additives which include but are not limited to antioxidants, extreme pressure (EP) agents, wear reduction agents, viscosity index improvers, anti-foaming agents, mixtures thereof and the like; and combination thereof. Component C can be used alone or in combination. In one embodiment Component C is at least one of an antioxidant and if component A is an antioxidant they are not the same antioxidant.

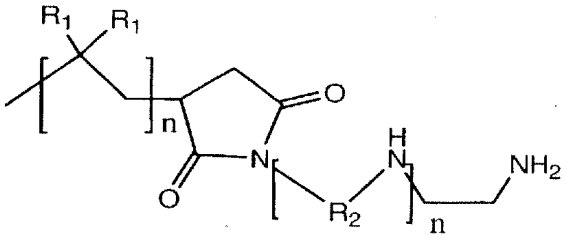
The gel contains component A in the range of about 0.1 % to about 95 %, in one embodiment about 5% to about 70% and in another embodiment about 7% to about 50% of the gel. The gel contains component B in the range of about 0.1% and about 99%, in one embodiment about 5% to about 80% and in another embodiment about 10% to about 70% of the gel. The gel contains component C in the range of about 0% to about 95%. In one embodiment about 1% to about 70% and in another embodiment about 5% to about 60% of the gel.

In accordance with the present invention the gel formed is an oil based gel. The gel is selected from the group comprising at least one of dispersants, dispersant precursors (such as alkyl or polymer succinic anhydrides) detergents, antioxidants, and mixtures thereof. Optionally, soluble additives may be added to the gel as desired, in particular oil soluble lubricating additives. The additives include, but are not limited to antioxidants, friction reducing agents, extreme pressure (EP) agents, wear reduction agents, viscosity index improvers, anti-foaming agents, anti-misting

combination. The dispersant is present in the range from about 0.1% to about 95% of the gel, preferably from about 1% to about 70% of the gel, and preferably from about 7% to about 50% of the gel.

The dispersant in the gel includes but is not limited to an ashless dispersant such as a polyisobutenyl succinimide and the like. Polyisobutenyl succinimide ashless dispersants are commercially-available products which are typically made by reacting together polyisobutylene having a number average molecular weight ("Mn") of about 300 to 10,000 with maleic anhydride to form polyisobutenyl succinic anhydride ("PIBSA") and then reacting the product so obtained with a polyamine typically containing 1 to 10 ethylene diamine groups per molecule.

Ashless type dispersants are characterized by a polar group attached to a relatively high molecular weight hydrocarbon chain. Typical ashless dispersants include N-substituted long chain alkenyl succinimides, having a variety of chemical structures including typically:



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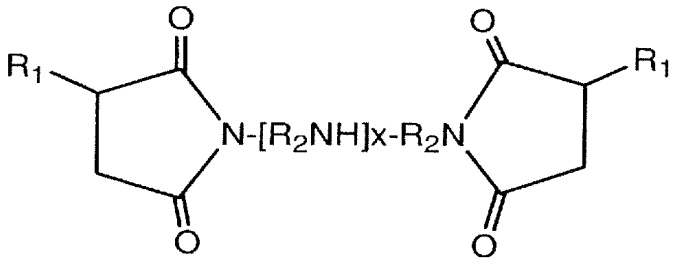


wherein each R^1 is independently an alkyl group, frequently a polyisobutyl group with a molecular weight of 500-5000, and R^2 are alkenyl groups, commonly ethylenyl (C_2H_4) groups. Succinimide dispersants are more fully described in U.S. Patent 4,234,435 which is incorporated herein by reference. The dispersants

The detergents include but are not limited to overbased sulfonates, phenates, salicylates, carboxylates, overbased calcium sulfonate detergents which are commercially-available, overbased detergents containing metals such as Mg, Ba, Sr, Na, Ca and K and mixtures thereof and the like. The detergents may be used alone or in combination. Detergents are described, for example, in U.S. Patent 5,484,542 which is incorporated herein by reference. The detergents are present in the range from about 0.1% to about 99%, preferably from about 5% to about 80% and more preferably from about 10% to about 70% by weight of the gel.

Antioxidants include but are not limited to alkyl-substituted phenols such as 2, 6-di-tertiary butyl-4-methyl phenol, phenate sulfides, phosphosulfurized terpenes, sulfurized esters, aromatic amines, diphenyl amines, alkylated diphenyl amines and hindered phenols.

The antioxidant includes amine antioxidants and is not limited to bis-nonylated diphenylamine, nonyl diphenylamine, octyl diphenylamine, bis-octylated diphenylamine, bis-decylated diphenylamine, decyl diphenylamine and mixtures thereof.

The antioxidant includes sterically hindered phenols and includes but is not limited to 2,6-di-tert-butylphenol, 4-methyl-2,6-di-tert-butylphenol, 4-ethyl-2,6-di-tert-butylphenol, 4-propyl-2,6-di-tert-butylphenol, 4-butyl-2,6-di-tert-butylphenol 2,6-di-tert-butylphenol, 4-pentyl-2,6-di-tert-butylphenol, 4-hexyl-2,6-di-tert-butylphenol, 4-heptyl-2,6-di-tert-butylphenol, 4-(2-ethylhexyl)-2,6-di-tert-butylphenol, 4-octyl-2,6-di-tert-butylphenol, 4-nonyl-2,6-di-tert-butylphenol, 4-decyl-2,6-di-tert-butylphenol, 4-undecyl-2,6-di-tert-butylphenol, 4-dodecyl-2,6-di-tert-butylphenol, 4-tridecyl-2,6-di-tert-butylphenol, 4-tetradecyl-2,6-di-tert-butylphenol, methylene-bridged sterically hindered phenols include but are not limited to 4,4'-methylenebis(6-tert-butyl-o-cresol), 4,4'-methylenebis(2-tert-amyl-o-cresol), 2,2'-methylenebis(4-methyl-6-tert-butylphenol), 4,4'-methylene-bis(2,6-di-tertbutylphenol) and mixtures thereof.

Another example of an antioxidant is a hindered, ester-substituted phenol, which can be prepared by heating a 2,6-dialkylphenol with an acrylate ester under

methacrylate are examples of nitrogen-containing monomers and the like. Polyacrylates obtained from the polymerization or copolymerization of one or more alkyl acrylates also are useful as viscosity modifiers. The viscosity modifiers may be used alone or in combination.

Functionalized polymers can also be used as viscosity modifiers. Among the common classes of such polymers are olefin copolymers and acrylate or methacrylate copolymers. Functionalized olefin copolymers can be, for instance, interpolymers of ethylene and propylene which are grafted with an active monomer such as maleric anhydride and then derivatized with an alcohol or an amine. Other such copolymers are copolymers of ethylene and propylene which are reacted or grafted with nitrogen compounds. Derivatives of polyacrylate esters are well known as dispersant viscosity index modifiers additives. Dispersant acrylate or polymethacrylate viscosity modifiers such as Acryloid™ 985 or Viscoplex™ 6-054, from RohMax, are particularly useful. Solid, oil-soluble polymers such as the PIB, methacrylate, polyalkylstyrene, ethylene/propylene and ethylene/propylene/1, 4-hexadiene polymers, can also be used as viscosity index improvers.

The viscosity modifiers are known and commercially available. The viscosity modifiers are present in the ranged about 0% to about 20%, preferably about 5% to about 15% and more preferably about 7% to about 10% of the gel.

Optionally, an inert carrier can be used if desired. Furthermore, other active ingredients, which provide a beneficial and desired function to the soot being decreased, can also be included in the gel. In addition, solid, particulate additives such as the PTFE, MoS_2 and graphite can also be included.

In an embodiment of this invention, the internal combustion engine is equipped with an exhaust after-treatment device. Exhaust after-treatment devices are used for modern engines to meet the new low exhaust emission standards. These systems are used to reduce undesirable emissions in the exhaust gases of internal combustion vehicle engines and are located in the exhaust system connected to the engines.

In one embodiment of this invention, catalysts are employed in the exhaust systems of internal combustion engines to convert carbon monoxide, hydrocarbons

Specific Embodiments

Examples:

In order to more thoroughly illustrate the present invention, the following examples are provided.

A. Gel Preparation

A representative gel, known as Composition X is prepared by first mixing components A and C, and then adding component B with mixing in the proportions listed below. The resulting mixture is heated at 120° overnight to produce the final gel.

Component	Chemical Description	% wt of Composition X
A	Polyisobutenyl (2000 Mn) succinimide Dispersant	20%
B	400 TBN Overbased Alkylbenzenesulfonate Deteregent	60%
C	Nonylated Diphenylamine Antioxidant	20%

B. Fleet Test

Test Vehicles

The test involved two trucks. Each truck uses two full-flow oil filters.

Test Filters

For the experiment runs, each engine was equipped with a filter with one cup into which was placed 400 g of Composition X additive gel and placed at the bottom of the filter. In the comparative runs, the same filter was used without additive gel in the cup. The additizing cup had twelve of 1/4" diameter diffusion holes at the top of the cup above the surface of the gel for 13 experiment – 34 experiment runs (Tables 2 and 3) and twelve of 1/16" diameter diffusion holes for experiment runs 1-12 (Table 1).

Test Oil

A 15W40 fully qualified (SAE-CI-4) oil was used in this test.

Test Procedure

The test vehicles was operated for 4 runs: 1) a baseline with standard filters, 2) a test run with two large hole cup filters on Truck #1 and two small-hole filters on Truck #2, 3) a second test run with two large hole cup filters on Truck #2 and two small-hole filters on Truck #1, and #4) a repeat baseline. For each run, both filters was

Table 1. Truck 1 Comparative (w/o gel) and Experimental (w/gel) Runs

Experiment Number	Vehicle #	Oil Miles	% Soot	Vis100	Experiment Number	Vehicle #	Oil Duration	% Soot	Vis100
1 Comp	1	0	0.00	14.88	1 Exp	1	0	0.10	14.42
2 Comp	1	554	0.10	13.69	2 Exp	1	573	0.20	13.42
3 Comp	1	1,038	0.20	13.41	3 Exp	1	1,069	0.10	13.09
4 Comp	1	2,349	0.30	12.78	4 Exp	1	2,754	0.20	12.38
5 Comp	1	5,147	0.90	12.42	5 Exp	1	5,279	0.30	11.87
6 Comp	1	7,638	1.30	13.00	6 Exp	1	7,408	0.60	11.60
7 Comp	1	9,616	1.60	13.99	7 Exp	1	9,668	0.80	11.85
8 Comp	1	12,861	2.20	12.30	8 Exp	1	12,818	0.90	13.64
9 Comp	1	14,740	2.20	12.32	9 Exp	1	15,831	0.90	12.79
10 Comp	1	17,239	2.40	12.46	10 Exp	1	18,306	1.00	11.80
11 Comp	1	19,482	2.70	12.35	11 Exp	1	20,173	1.20	11.87
12 Comp	1	22,204	3.00	12.43					

Table 2. Truck 2 Comparative (w/o gel) and Experimental (w/gel) Runs

Experiment Number	Vehicle #	Oil Duration	% Soot	Vis100	Experiment Number	Vehicle #	Oil Duration	% Soot	Vis100
13 Comp	2	0	0.00	14.88	13 Exp	2	0	0.10	13.97
14 Comp	2	507	0.10	13.97	14 Exp	2	550	0.10	13.76
15 Comp	2	986	0.20	13.29	15 Exp	2	1,024	0.10	13.11
16 Comp	2	2,645	0.20	12.92	16 Exp	2	2,399	0.10	12.58
17 Comp	2	5,083	0.60	12.46	17 Exp	2	4,375	0.20	17.24
18 Comp	2	6,982	0.90	12.08	18 Exp	2	7,051	0.40	11.88
19 Comp	2	9,539	1.30	10.90	19 Exp	2	9,728	0.70	11.63
20 Comp	2	11,712	1.60	12.16	20 Exp	2	12,036	0.80	11.76
21 Comp	2	14,209	1.70	12.05	21 Exp	2	14,904	1.00	11.74
22 Comp	2	16,714	1.80	12.35	22 Exp	2	18,129	0.90	11.97
23 Comp	2	19,048	2.10	14.32	23 Exp	2	20,224	1.10	12.02

C. **GM 6.5L Engine Test**

Test Engine

GM 6.5L Engine see ASTM D5966.

Test Filters

For the Exp runs, each engine was equipped with a filter with one cup into which was placed 400 g of Composition X additive gel and placed at the bottom of the filter. In the comparative runs, the same filter was used without additive gel in the cup. The additizing cup had twelve of 1/4" diameter diffusion holes at the top of the cup above the surface of the gel.

Test Oil

A 15W40 fully qualified (SAE-CI-4) oil was used in this test.

Procedure

See Designation: D 5966 – 99 "Standard Test Method for Evaluation of Engine Oils for Roller Follower Wear in Light-Duty Diesel Engine 1, AMERICAN SOCIETY FOR TESTING AND MATERIALS, 100 Barr Harbor Dr., West Conshohocken, PA 19428, from the Annual Book of ASTM Standards. Copyright ASTM.

Results

The results are shown in Table 4, 35comparatives - 37comparatives is for comparative runs with no additive in the filter, experiments 35 experimental – 36 experimental are for filters with gel. Table 5 summarizes experiments in which the antioxidants withheld from the gel (37 Experimental) compared to baselines (37Comparatives). Table 6 shows soot production with no gel in the filter, with and without dosing of a 1:1 mixture of antioxidant:dispersant throughout the 50 hr test. These data show that antioxidant and dispersant do not have to be added from the gel, but dosing of these components by other means also results in reduced soot levels in the engine oil.

D. Mack T-8 Engine Test

Test Engine

Mack T-8 Diesel Engine.

Test Filters

For the experiment runs, the engine was equipped with an oil pan with a 1" deep tray, into which was placed 400 g of Composition X additive gel. In the comparative runs, an oil pan without additive was used.

Test Oil

A 15W40 fully qualified (SAE-CI-4) oil was used in this test.

Procedure

A Short T-8 test was used. The Short T-8 is a modified version of the T-8/T-8E ASTM test. Conditions are shown below:

Speed (rpm): 1800 Fuel Flow (kg/hr): 63.3 Intake Manifold Temp. (C): 43 Coolant Temp. (C): 85 Crankcase Pressure (kPa): .25-.75

Inlet Air Restriction (kPa): 2.25-2.75 Exhaust Back Pressure (kPa): 3.1 Engine Timing (BTDC): 15 degrees

The engine timing corresponds to an average soot production rate in the Comp experiment of 0.006%/hour in a 7 quart oil sump.

Results

The results are shown in Table 7, Experiments 39 Comparatives and 39 Experimental, and in Figure 1.

From the above description and examples of the invention those skilled in the art will perceive improvements, changes and modifications in the invention. Such improvements, changes and modifications within the skill of the art are intended to be covered by the appended claims.

9. The composition of claim 8 wherein the total base number (TNB) of the overbased detergent is in the range from about 100 to about 400.

10. The composition of claim 2 when the dispersant is selected from the group comprising ashless succinimide, polyisobutenyl succinimide, substituted long chain alkenyl succinimides, high molecular weight esters, mannich dispersants, N-substituted long chain alkenyl succinimides, carboxylic dispersants, amine dispersants, polymeric dispersants, decyl methacrylate, vinyl decyl ether, aminoalkyl acrylates, acrylamides, poly-(oxyethylene)-substituted acrylates, high molecular weight olefins with monomers containing polar substitutes and a mixtures thereof; and a detergent selected from the group comprising overbased sulfonates, phenates, salicylates, carboxylates, overbased calcium sulfonate detergents, overbased detergents containing metals such as Mg, Ba, Sr, Na, Ca and K and mixtures thereof; and an antioxidant selected from the group comprises alkyl-substituted phenols, 2, 6-di-tertiary butyl-4-methyl phenol, phenate sulfides, phosphosulfurized terpenes, sulfurized esters, aromatic amines, diphenyl amines, alkylated diphenyl amines, hindered phenols, bis-nonylated diphenylamine, nonyl diphenylamine, octyl diphenylamine, bis-octylated diphenylamine, bis-decylated diphenylamine, decyl diphenylamine, 2,6-di-tert-butylphenol, 4-methyl-2,6-di-tert-butylphenol, 4-ethyl-2,6-di-tert-butylphenol, 4-propyl-2,6-di-tert-butylphenol, 4-butyl-2,6-di-tert-butylphenol 2,6-di-tert-butylphenol, 4-pentyl-2,6-di-tert-butylphenol, 4-hexyl-2,6-di-tert-butylphenol, 4-heptyl-2,6-di-tert-butylphenol, 4-(2-ethylhexyl)-2,6-di-tert-butylphenol, 4-octyl-2,6-di-tert-butylphenol, 4-nonyl-2,6-di-tert-butylphenol, 4-decyl-2,6-di-tert-butylphenol, 4-undecyl-2,6-di-tert-butylphenol, 4-dodecyl-2,6-di-tert-butylphenol, 4-tridecyl-2,6-di-tert-butylphenol, 4-tetradecyl-2,6-di-tert-butylphenol, 4,4'-methylenebis(6-tert-butyl-o-cresol), 4,4'-methylenebis(2-tert-amyl-o-cresol), 2,2'-methylenebis(4-methyl-6-tert-butylphenol), 4,4'-methylene-bis(2,6-di-tertbutylphenol) and mixtures thereof. .

11. A process comprising contacting a portion of the engine oil with a gel of the composition of claim 3 resulting in the reduction of soot in the engine oil and/or emissions in an engine exhaust.

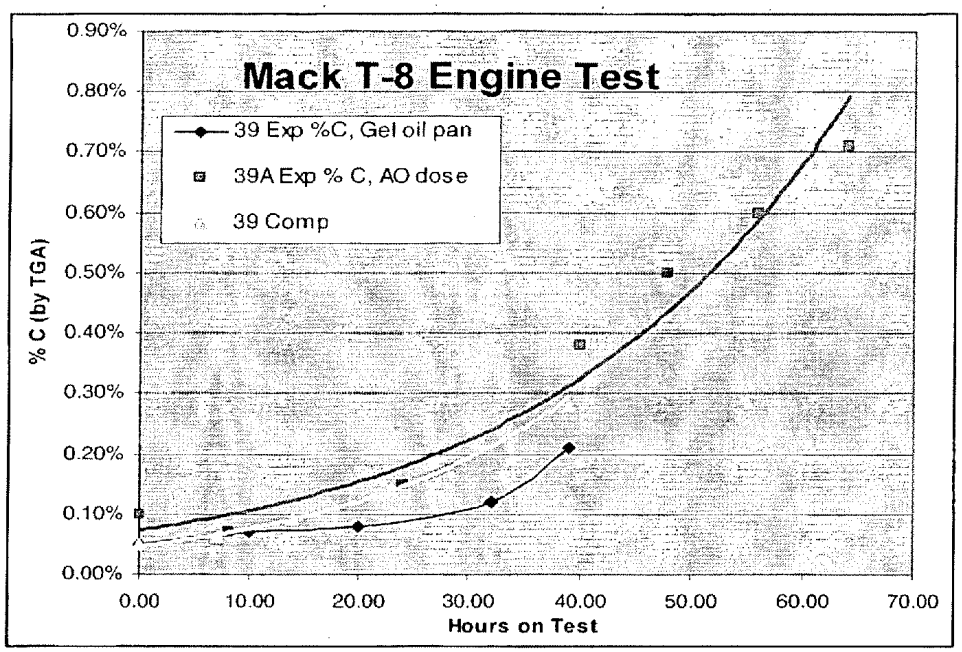
12. The process of claim 11 wherein the gel is positioned to contact the oil

soot-reducing gel wherein the gel comprises a dispersant, a detergent, an antioxidant and combinations thereof and results in the reduction of one of the following from an engine soot, emission or combinations thereof.

24. A gel containment device for an engine oil lubricating system comprising a housing and a container with a gel, and wherein the gel comprises a dispersant, a detergent, an antioxidant and combinations thereof for the soot reduction, emissions reduction or combinations thereof of an engine.

FIGURE 1

Mack T-8 Engine Test Soot production Comparisons



INTERNATIONAL SEARCH REPORT

PCT/US2004/026884

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C10M163/00 C10M175/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2004/014614 A1 (BURRINGTON JAMES D ET AL) 22 January 2004 (2004-01-22) paragraphs '0015! - '0018!, '0025!, '0030! - '0040!, '0042!, '0048!; examples	1-24
E	WO 2005/003265 A (THE LUBRIZOL CORPORATION) 13 January 2005 (2005-01-13) claims 1-21; examples	1-24
E	WO 2005/003266 A (THE LUBRIZOL CORPORATION) 13 January 2005 (2005-01-13) claims 1-18; examples	1-24

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
29 April 2005	06/05/2005

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Kazemi, P